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Interaction of laser beams with pure PMMA and with inclusion/impurities/dopants for various applications

ABSTRACT

PMMA (Polymethylmethacrylate), as one plastic material with pretty well corrosion resistance, is widely used and investigated both from theoretical and engineering aspects. When quantum generators in visible range were invented in microwave range (in 6th decade of the last century), they were called ''a solution looking for a problem''. Lasers are now ubiquitous, with compact disk players and eye surgery vying as the most familiar applications. Interaction of lasers in various working regimes and wavelengths with PMMA material were for a long time the object of investigation. In presented paper experimental part of investigation was done as well as simulations of temperature distribution.

Specific attention is paid to microstructure of damaged surfaces quantitatively analyzed by image processing programs. Heat treating with lasers allows selective surface hardening against wear with little or no distortion of the component. Because this eliminates much part reworking that is currently done, the laser system's capital cost is recovered in a short time. One consideration crucial to the success of a heat treatment operation is control of the laser beam irradiance on the treated surface. The optimal irradiance distribution is driven by the thermodynamics of the laser-material interaction and by the sample/component geometry. Typically, irradiances between 500-5000 W/cm^2 satisfy the thermodynamic constraints and allow the rapid surface heating and minimal total heat input required.

Key words: *PMMA, laser modification, image analysis, modeling, temperature distribution, simulations*

1. INTRODUCTION

Problems about interaction of laser beam with material, destruction or modulation of surface are analyzed from many aspects. Such problems could be considered from the theoretical models, when the phase boundaries may be formed during transformation and still, they are not well known; after all of possible transformations the destruction could be happened. Practically, it is possible to modify the surface properties (optical, technological and others), and to change the chemical composition on defined depth. Interactions also could be monitored as a set of parameters during engraving, microhardness changes, surface roughness, thermal, acoustic or other properties. With ultrafast lasers applying, some kinds of thermal modeling should be provided only by using of an electron part of physical properties [1-18].

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The problems of reliability, stability and repeatability when transparent optical materials were destroyed are particularly *intrigant*. There is a need for exact definition of the *transparency*, keeping in mind that nonlinear behavior is achieved, but also in a wide spectral range of various lasers. The simulation of interaction is based on data of thermal nature, optical nature (reflectivity, absorption, refraction index) and all of those properties are dispersive (depend on the wavelength), incident angles of applied beams for material processing, material purity and others. At semiconductors, could be induced interactions as metallic or dielectric type [13,14].

Interaction of laser beams in various energy ranges and exposition time is of particular interest. Experimental and theoretical interaction's analyses of chosen laser types with transparent materials here will be analyzed and discussed.

Fundamental significance in research of disintegration mechanisms is the investigation of the dependence of damaged shape versus the energy density/power. Characteristics thresholds for many different processes were defined and they characterized specific transformation up to disintegration. Many of them are characterized not only with transformation liquid-solid-gas/vapor but including: plasma phenomena, ejected mass, emission of different particles, acoustic effects, amorphous-crystal transformations, chemical concentration changes, etc. [18].

Classical scheme of interaction laser-material could be find in various references. Crossing on higher intensities many conclusions overturned, and also *classical* boundaries metal-nonmetal, semiconductor, crystal-amorphous state, *are modified*. The particular place in interactions between high energy beams (*elion* techniques) find out the interaction of material with laser beams. The fundamental significance in investigation of mechanisms of destroying plays for the optical media and dimensional dependence between energy density – intensity, that is strongly related to the minimal beam sections [19-23].

Materials which belong to the scintillator type are very important as active material for lasers-quantum generators, too. In the *past* they were almost important only for nuclear detection and fluorescent phenomena. In the systems of nuclear detectors of organic material, scintillators are in some vases covered with with specific transparent material or metallic protection coatings depending on the energy of investigated radiations. For optical photons, coatings (the protections) should to be transparent for optical (VIS/IR/UV) radiation depending on the chosen photocathode [24-25].

The hygroscopy of scintillator window is particularly present at NaJ (Ta), which is enveloped in aluminium foil. Some of our experiments were organic and plastics scintillators, or similar material, too. Some of them were with TEA CO_2 ,Nd^{3+}:YAG, Cr^{3+}:Al_2O_3, alexandrite, semiconductor-diode or other laser types. The incident laser beams were directed by using mirror and focused by lens, depending of the laser wavelength (SnSe – for CO_2 laser). Depending on the laser working regimes, (cw or pulsed) energy, should be monitored through appropriate detectors. To illustrate the operation parameters for our expositions for many materials we will presented some of them: 1mJ multimode; 0.7 TEM_{00}, divergency, 3mrad. In the case of TEA CO_2 laser, the first part of the complex pulse of 40ns contained 35-50% of energy: working repetition frequency was 0-2Hz or more higher as well as the number of pulses in the same points. The parameters for gas laser were changeable with different relations of gas mixture: some were He:CO_2:N_2 respectively 5.9:1.4:1.3 ft^3/h. The optical gratings on exit were 150 lin/mm [25], and tuning was possible from 9.6-11.2μm and for Nd^{3+}:YAG was possible to use higher harmonics beside ground transition. All scintillators, plastics, glass or polymer components were intensively damaged by applied working parameters.

2. EJECTED MATERIAL

The question of ejected mass begins with considering the absorption of laser beam into defined material. Obtained results serve for more precise interpretation of phenomena and processes during exposition to laser beams and for quantification of such dependencies. The depth of laser beam penetration into metals frequently is on level »10^{-5}cm, but at organic materials this depth is greater, as the mechanisms of energy transformation are different. The interaction of electrons with crystal lattice produces increasing increasing the temperature (if for such processes are valid thermal models with various approximation and modifications) [27-29].

Time of energy exchange during collision is » 10^{-12}-10^{-14}s. Relaxation times give the way for modeling where ultra fast laser pulses are given in exposition parameters. The transformations of absorbed energy into heat is conducted almost immediately, it is concluded on the fact that the temperature near interaction zone could grow fast. The temperature distribution could be modeled and on that base is possibly to *read out* the temperature in details. At these circumstances, the relationship between LAZ (Laser Affected Zone) and HAZ (Heat Affected Zone) becomes important [29]. Those zones could be distinguished during interactions with cw lasers, laser in free generation regime, Q-switch/spoiling or lasers with ultra short pulses. Commonly could be found simulations and analytical models for Gaussian distribution, with the exact value of temperature at the focus center, which depends from P_0-total energy of absorbed radiations, energy at the focal*point*, estimated radii of the estimated temperature distribution, specific heat of material, material density, and coefficient of temperature conduction.

Except for experimental approximations, the analytical expressions were disagreed, so other expressions should be used, with other kinds of parameters. For the initial coefficient of reflection in linear area, it plays an important role; for nonlinear processes has to be taken into account the dependence R (I). The temperature increasing at surface provokes the decreasing of the reflection coefficient R. It exists by small intensities, i.e. by small power densities (10^7W/cm^2) and high (10^{10}W/cm^2) densities in focus. The low losses exist also due to thermoelectric emission and reemission from the hot surface. These processes are negligible in the case of high-power densities.

The obtained expressions (developed) are for the case of the moving source with different dynamics, but they are generally for Gaussian beam profile shapes. However, all laser energy does not spend for the temperature increasing. Often, great part is reflected from the surface. An inert, absorbent coating for laser heat treatment has also been developed that eliminates the fumes generated by conventional paint coatings during the heat-treating process with CO_2 laser beams.

In relation to the interaction of laser beam, there should be done a connection with classical theories about linear viscosity from 1953, which is limited on lower levels, but it is worth to know that plastic materials show nonlinear viscous -elastic behavior, even at low stress levels during short loading times [11]. It was an intention to obtain the constitutive relation between loads and stress field at a high loads.

Until now, a lot of models were stated; according to [11] such models could be divided into three groups: engineering model, rheological and modular. For describing the behavior in high stress field for short time, the attention is focused on rheological models.

The rank of application for many models is limited, because the engineering models offer some *rougher* limitations versus creep, recovery and behavior under complex model of loading.

Rheological models include a few approaches, but schappery processes were chosen for description of creep and recovery tests for nonlinear viscous-elastic behavior of PMMA. Other models are considered to be more specific [11].

The most frequent objects of investigations are: forming of microstructures (3D) at laser ablations [30], frequency conversion at subnano joule fs laser pulses, miscibility of PMMA with various inclusions and in relation to Vicat temperature, micromachining with CO_2 and back-end processing for rapid working processes of PMMA based microfluidic systems [31, 32]. Vicat temperature is one of the parameters that determines the laser cutting-ability, e.g. efficiency of laser processing, which is presented through laser number N. According to N, a categorization presents which materials are easier or heavier processed by laser techniques [29, 32].

The aim was to compare the type and mechanism of a fracture with the corresponding type of theoretical prediction expressed through the modulus of elasticity of polymeric composites, and other mechanical magnitudes such as Poisson ratio, etc.

The modulus of elasticity is an important mechanical characteristic of structural material necessary for the estimation of the mechanical characteristics of the construction and for their final testing. Since in references only approximate values of the parameter which expressed the dependence of polymeric composites versus load can be found, than further investigations might be provided. Therefore of importance is to compare reliable theoretical prediction based on the knowledge of composition and modulus of compounds. These methods were experimentally tested somewhere in references.

The sintering technologies should be up today investigated by using the various initial material parameters to final sintered products[26]. It means the shape of the particles (spherical or other), achieving the density similar to theoretical density, dependence of properties from temperature and pressure during processing, etc, are of constant interest. Some facts could be found as unsolved: some microscopic analyses show the distinguishing from Frenkelj´s kinetic equation and contact surfaces (interface). The analysis of the sintering methods for spherical particles and comparison with simulations, dependence on temperature and sintering pressure and discussion versus Frenkelj's kinetic equations are performed, too. The rheologic behavior of polymers is specific in relation viscoelastic-viscous state. The changes were monitored by circles, especially when contact circles undergo to moving and their centers became closer. The degrees of porosity exist in three phases: open, open to closed, and spheroidization. Frenkelj's activation energy is similar to viscosity processes and sintering through those mechanisms. Thermal, thermo-mechanical and structural characterization of incident materials include hydrodynamic and kinematics formation processes, thermal transfer and analyses of obtained models should include coefficients according to variation of radii and direction of uniform characteristics. Of interest is also geometrical structure, physical, mechanical, chemical performances, rupture magnitudes. X-ray diffraction has shown absence ofcrystallity, but also birefringence. The temperature increasing is affected optical anisotropy (i.e. the anisotropy will be vanished).

Mathematical model of fibre forming enables the monitoring of the radii/reaction stability in the system. The discrepancy from nominal values of 1D model is a base for more complex simulation of fibre formation, with taking account the real temperature gradients at fibre drawing, and other production variables. Although PMMA as one of very investigated material, it is today still topics from many fields in linear and nonlinear cases [34-41].

3.SIMULATION OF INTERACTION OF SELECTEDMATERIALS AND LASER REGIME. DETERMINING TEMPERATURE DISTRIBUTION

3.1. Temperature simulation after laser treating ofPMMA

Simulations for PMMA, here as one of the of plastic materialsrepresentative, in the regime of ms-laser impulses were conducted by the Comsol program package for chosen samples with diameter of 80mm and thickness of 5mm. The parameters important for simulation were adopted based on references [6]. Thermal conductivity(λ), density (ρ_0) and specific heat (C_P,) in simulation processes were used as constant (in applied laser beam parameters), so their values are 0.18 W/mK, 1190kg/m^3 and 1450 J/kg K in SI units, respectively. Used laser parameters are: energy 4J, pulse width 4ms, i.e. power 1kW and diameter of beam 2mm.

Unlike in some previous simulations [17.23] when time step was constant, in presented case was a variable with small steps. Therefore, more detailed image of laser heating up by applied laser parameters and shortly after laser exposition was given. At the first interval, till the time of 40 ms, steps of 1ms are used; in interval 40-500ms bigger steps of 10ms were adopted.

So, simulation was made in steps 1ms steps for 40ms and from 40 to 500ms at increments of 10ms. The graphics are presented in four specific moments: 10, 40, 100 and 500ms, following the dynamics of heating and cooling. The pictures were marked with e.g. PMMA500ms to indicate a situation in the temperature field at 500ms.

The results or performed simulations are presented in the figures from 1 to 4.

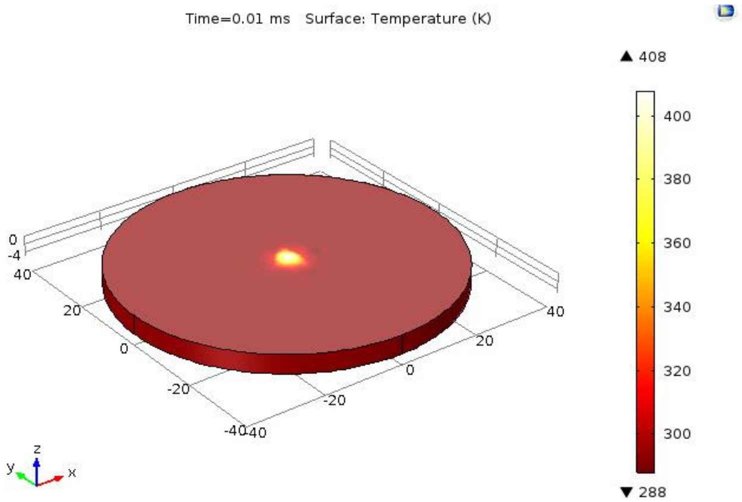


Figure1.Temperature simulation for PMMA at 10ms

sl. 1. Simulacija temperature za PMMA pri 10ms

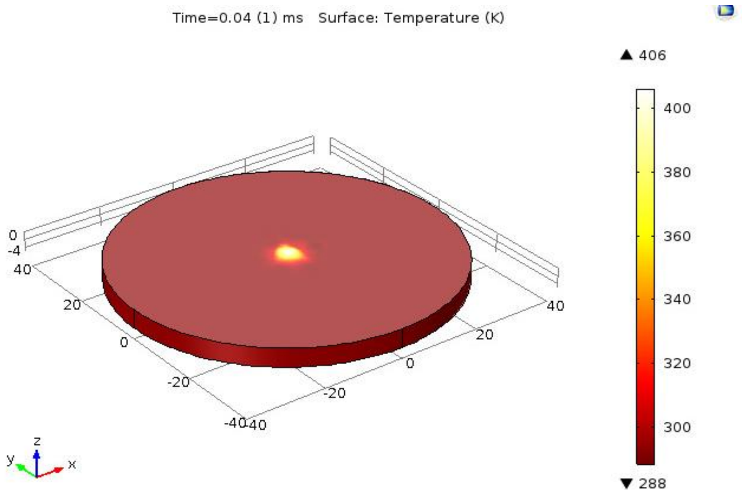


Figure2.Temperature simulation for PMMA at 40ms

sl. 2. Simulacija temperature za PMMA pri 40ms

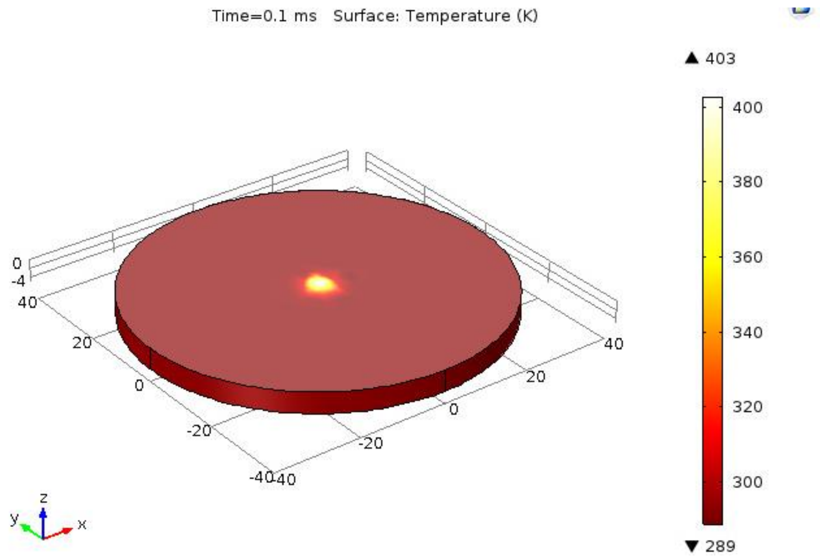


Figure 3.Temperature simulation for PMMA at 100ms

sl. 3. Simulacija temperature za PMMA pri 100ms

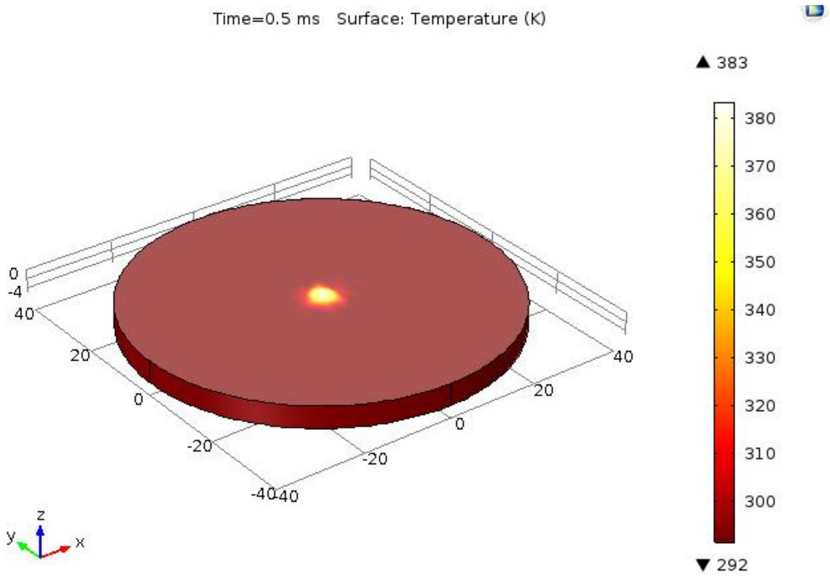


Figure 4.Temperature simulation for PMMA at 500ms

sl. 4. Simulacija temperature za PMMA pri 500ms

3.2. Light microstructure analysis after drilling, cutting and engraving operations

Different types of lasers were used to investigate their influence on samples made from various materials: PMMA, glass, ceramics, metals and thin films, in versatility of geometric shapes. In this paper main subject is material based on PMMA. Quantitative analysis by using appropriate image program packages, here Image J, was performed on micrograph from the series I in provided analysis. Micrographs for this analysis were obtained using light microscope at magnification x 100. The given analyses presents the methods by which performed interaction is studied.

a) Light microscopic analysis

Damaged samples were analyzed by light microscope Litz MZ6 and SEM, as presented in figs. 5÷8). Changes in microstructure induced by laser power fluctuations are presented in Fig. 5, for another details see [7].

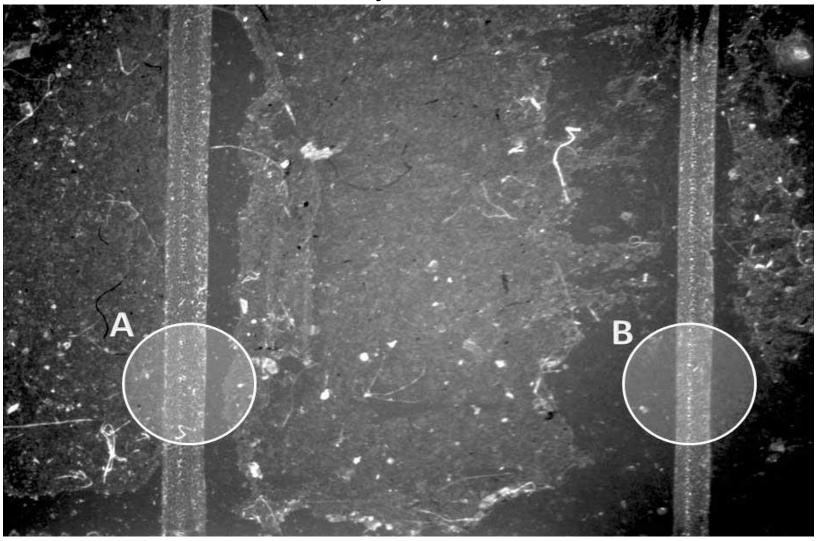


Figure 5. Analysis by light microscope, x20; A-chanel obtained with regular input of energy/voltage supply and B-channel obtained when the fluctuatuion of voltage input was -10%

The areas near the cutting edge, near cutting channel and crater bottom as well as crater walls, and ejected material close to the laser damages were the object of provided investigations.

Figure 6. Analysis by light microscope at cutting channel, x60

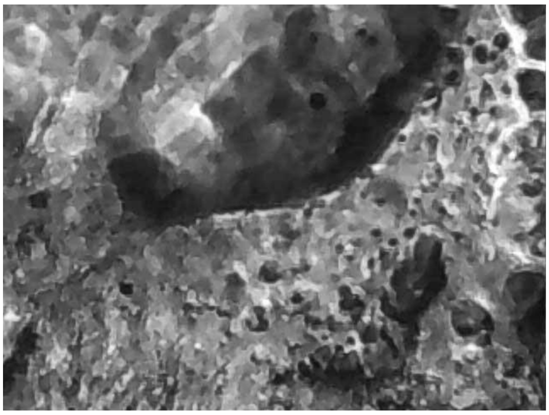
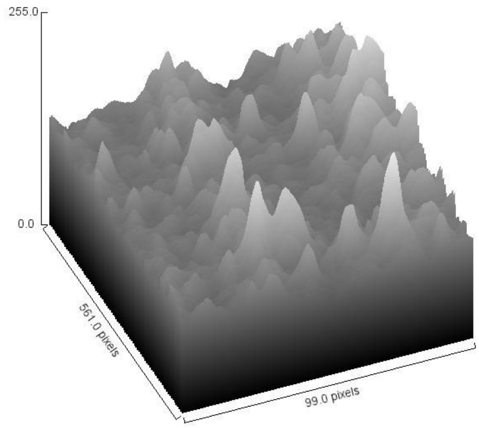


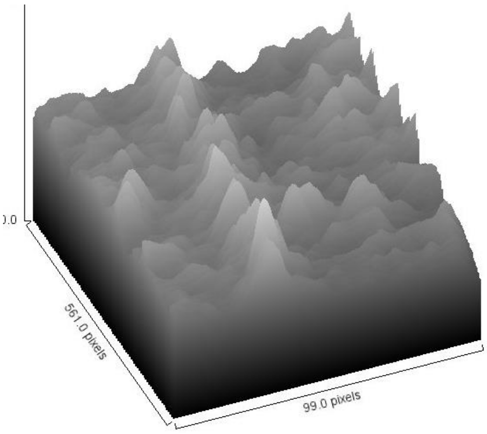
Figure 7. Analysis by light microscope at channel line,x60

b) morphological analysesanalysis of PMMA after laser treating

The upper part of the Fig.5. is used for further analyzing and given in Fig. 8.



a)



b)

Figure 8.Roughness analysisfrom Fig. 5.: a)position A and b) position B b)x100

The extracted area has surface of 63309 pixels. Therefore, the medium level of *greyiness* of the analyzed area is 160.840 (the range is from 0 to 255), with standard deviation 60.329. The minimum value of the shades of gray does not fall below 10 in isolated areas.

From the obtained gray level measurement, grey level is slightly higher 8.4% (gray level difference: 174.345) than in extracted areas. That fact is expected, because of darker shades that are characterized by induced damage. The term laser damage threshold is not exactly treated in references always, and it could be object of particular attention. The obtained standard deviation is higher than previous mentioned, because unexposed area (of different sizes) is included in analysis; therefore, microstructure looks more uniform, it is characterized quantitatively by 65.203 pixels; it means 8.1% is higher. An interesting coincidence in differences of standard deviations and medium levels (8.4% and 8.1%), appeared, and such behavior needs additional study and detailed analysis.

Fig. 9.is modified (in pseudo color and contrast) in order to the better visualization of the damaged surface structure of the treated sample. These modifications are preferable made for correct quantitative image analyses, Fig. 9.

Figure 9. Modified image of Fig. 5. for easier detection of damaged structure in the quantification process.

4. DISCUSSION AND CONCLUSIONS

In our works, we used various types of lasers in various working regimes, i.e. dynamic modes of their active materials. Explicitly, we have worked with Nd^{3+}:YAG, Cr^{3+}; Al_2O_3, and CO_2 lasers mostly. We also used semiconductor lasers with 400nm. Pure PMMA and PMMA with pigment were exposed to laser beams in various regimes: free generation regimes, Q switch to mono and multi-pulse regimes of mentioned lasers and their harmonics (1064, 532, 266, 300nm). In experiments and in simulation processes it is shown that crystals (linear and nonlinear) have different behavior than PMMA materials. Thermal process (cooling) was slower. Therefore, dynamics of thermals heating and cooling through and after interaction of PMMA is specifically and depend on applied wavelength.

The material properties, explained in references by using of storage modulus, loss modulus and glass transformation temperature (T_g) for polymer composites [11], are growing depending as the dopant concentration increases. The influence of nano particles could be of interest for further investigations.

Relation between temperature and Vickers hardness relatively is close to linear. Mechanical, i.e. dynamical, properties of nano composites depend on the composition, however. In literature could be found more data about transmittance, FTIR spectra, that are going to show the complementary data to the DMA results (temperature dependence of storage modulus, tangδ, loss modulus) changed with temperature increasing during the laser material interactions. Therefore, simulation and experiment should be compared and considered from different points of view including DMA (Dynamic Mechanical Analysis), DTA (Differential Thermal Analiyis) and others [11], to explain complex processes during PMMA – laser beam interactions, and responses to elastic/plastic loading regions.

Considering PMMA material, the behavior could be modeled close to theoretical approaches. The linear behavior of PMMA is throughly

investigated in references, especially at creep and recovery conditions. From uniaxial tensile creep and recoverytests is known that PMMA shows linear viscous behavior below 25MPa, but non linear behavior above this level of stress. At nonlinear range practically exist two subranges: first, from 25MPa-37.5 MPa, when the problem could be solved byseparation of variables $\varepsilon = f(\sigma)h(t)$,and second above 37.5 MPa, when the

relation ship is strongly nonlinear (prediction of separation of variables, coupling of stress and temporal intervals.

Discrepancies of data for damage thresholds of different types could be originated due to comparison of the results with no identical initial parameters which are unavoidable by estimation of boundary magnitudes. Therefore, the detailed experimental description is always needed (dependence of reflection coefficient on beam intensity, exact definition of thresholds for various processes, mean and maximal estimations). Note that term threshold must be carefully used with regard to procedure which is proposed 10-20 years ago. There is deeper treatment of discrepancies connected to statistics of modelling (Poisson, Gaussian, etc.). It seems that the number of data should be clearly declared. PMMA material is up to day very actual, due to large applications as pure or matrix for composites with many dopants.

The references include new recordings of transmissions in IR/VIS/UV ranges, that are independent of flux/ intensity, with linear (or more complex) behaviour under low and high beam intensities. FTIR spectra may give the answer to detailed vibration and should be responsible for interpretation the role of the dopant. The luminance processes in red portion of spectra increase with growing the concentration of definite dopant type (peaks at 611nm). Emission spectra are mixed in references with microhardness data. Strong dependence of temperature exists for storage modulus, loss modulus and tang δ for various percent of rare ions. Loss modulus and tang δ are characterized by some kinds of maxima values. The connection to fluorescence phenomena is intriguing (FTIR).

According to DMA analysis (difference mechanical analyses): E', E'' and corresponding T_g E' grows with dopant contents, as well as T_g, etc. The detailed correlation and fitting methods should be generally further discussed in regard to application linear or nonlinear dependences. Schapery's model as special case could be connected to creep under unaxial stress and reduced times. Therefore thermodynamic changes (g_o, g_1, g_2) are the function of applied stress. In linear range are thermodynamic changes related to the third order dependences versus Gibbs free energy (in relation to stress). Note that by laser beam are today the biggest produced pressures in laboratory environment and small local volumes in very short time. Due to those circumstances we think that expositions to laser beams in ato- and femto-second regimes could be obtained at least intuitive, experimental confirmation for material behaviour under high pressure.

For all of these cases interaction with lasers may be different in the power ranges, where the laser beam *recognize* the material. For general heat treatment, a uniform square or rectangular beam is one of the best options. For some special applications or applications where the heat treatment is done on an edge or corner of the sample/component, it may be better to have the irradiance decrease near the edge to prevent melting.

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IZVOD

INTERAKCIJA LASERSKIH ZRAKA SA ČISTIM PMMA I SA INKLUZIJAMA/NEČISTOĆAMA/DOPANTIMA ZA RAZLIČITE PRIMENE

PMMA (polimetilmetakrilat), kao jedan plastični materijal sa dobrom otpornošću na koroziju, široko se koristi i istražuje kako sa teorijskog, tako i sa inženjerskog aspekta. Kada su kvantni generatori u vidljivom opsegu izumljeni u mikrotalasnom opsegu (u 6. deceniji prošlog veka), nazvani su „rešenjem koje traži problem“. Laseri su sada sveprisutni, a kompakt disk plejeri i očna hirurgija se takmiče kao najpoznatije primene. Interakcija lasera u različitim režimima rada i talasnim dužinama sa PMMA materijalom dugo je bila predmet istraživanja. U predstavljenom radu je urađen eksperimentalni deo istraživanja, kao i simulacije raspodele temperature.
*Posebna pažnja je posvećena mikrostrukturi oštećenih površina kvantitativno analiziranih programima za obradu slike. Termička obrada laserima omogućava selektivno očvršćavanje površine od habanja sa malo ili bez izobličenja komponente. Pošto ovo eliminiše veliki deo prerade delova koji se trenutno vrši, kapitalni troškovi laserskog sistema se vraćaju u kratkom roku. Jedno od ključnih razmatranja za uspeh o*peracije termičke obrade je kontrola zračenja laserskog snopa na tretiranoj površini. Optimalna raspodela zračenja je određena termodinamikom interakcije lasera i materijala i geometrijom uzorka/komponente. Tipično, zračenja između 500-5000 W/cm2 zadovoljavaju termodinamička ograničenja i omogućavaju brzo zagrevanje površine i minimalan potreban ukupni unos toplote.*
Ključne reči: *PMMA, laserska modifikacija, analiza slike, modeliranje, raspodela temperature, simulacije*

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